(b) For each pair of measurements, determine the difference (discrepancy) between the candidate method measurement and reference method measurement. A discrepancy which exceeds the discrepancy specified in table C–1 of this subpart constitutes a failure. Figure C–1 of this subpart contains a suggested format for reporting the test results.

(c) The results of the first set of measurements shall be interpreted as follows:

(1) Zero failures. The candidate method passes the test for comparability.

(2) Three or more failures. The candidate method fails the test for comparability.

(3) One or two failures. Conduct a second set of simultaneous measurements as specified in table C–1 of this subpart. The results of the combined total of first-set and second-set measurements shall be interpreted as follows:

(i) One or two failures. The candidate method passes the test for comparability.

(ii) Three or more failures. The candidate method fails the test for comparability.

(4) For SO_2, the 1-hour and 24-hour measurements shall be interpreted separately, and the candidate method must pass the tests for both 1- and 24-hour measurements to pass the test for comparability.

(d) A 1-hour measurement consists of the integral of the instantaneous concentration over a 60-minute continuous period divided by the time period. Integration of the instantaneous concentration may be performed by any appropriate means such as chemical, electronic, mechanical, visual judgment, or by calculating the mean of not less than 12 equally spaced instantaneous readings. Appropriate allowances or corrections shall be made in cases where significant errors could occur due to characteristic lag time or rise/fall time differences between the candidate and reference methods. Details of the means of integration and any corrections shall be submitted.

(e) A 24-hour measurement consists of the integral of the instantaneous concentration over a 24-hour continuous period divided by the time period. This integration may be performed by any appropriate means such as chemical, electronic, mechanical, or by calculating the mean of 24 sequential 1-hour measurements.

(f) For ozone and carbon monoxide, no more than six 1-hour measurements shall be made per day. For sulfur dioxide, no more than four 1-hour measurements or one 24-hour measurement shall be made per day. One-hour measurements may be made concurrently with 24-hour measurements if appropriate.

(g) For applicable methods, control or calibration checks may be performed once per day without adjusting the test analyzer or method. These checks may be used as a basis for a linear interpolation-type correction to be applied to the measurements to correct for drift. If such a correction is used, it shall be applied to all measurements made with the method, and the correction procedure shall become a part of the method.

§ 53.33 Test procedure for methods for lead.

(a) *Sample collection.* Collect simultaneous 24-hour samples (filters) of lead at the test site or sites with both the reference and candidate methods until at least 10 filter pairs have been obtained. If the conditions of § 53.30(d)(4) apply, collect at least 10 common samples (filters) in accordance with § 53.30(d)(4) and divide each to form the filter pairs.

(b) *Audit samples.* Three audit samples must be obtained from the address given in § 53.4(a). The audit samples are 3/4×8-inch glass fiber strips containing known amounts of lead at the following nominal levels: 100 µg/strip; 300 µg/strip; 750 µg/strip. The true amount of lead, in total µg/strip, will be provided with each audit sample.

(c) *Filter analysis.* (1) For both the reference method samples and the audit samples, analyze each filter extract three times in accordance with the reference method analytical procedure. The analysis of replicates should not be performed sequentially, i.e., a single sample should not be analyzed three

times in sequence. Calculate the indicated lead concentrations for the reference method samples in µg/m³ for each analysis of each filter. Calculate the indicated total lead amount for the audit samples in µg/strip for each analysis of each strip. Label these test results as R_{1A}, R_{1B}, R_{1C}, R_{2A}, R_{2B}, ..., Q_{1A}, Q_{1B}, Q_{1C}, ..., where R denotes results from the reference method samples; Q denotes results from the audit samples; 1, 2, 3 indicate the filter number, and A, B, C indicate the first, second, and third analysis of each filter, respectively.

(2) For the candidate method samples, analyze each sample filter or filter extract three times and calculate, in accordance with the candidate method, the indicated lead concentrates in µg/m³ for each analysis of each filter. Label these test results as C_{1A}, C_{1B}, C_{2C}, ..., where C denotes results from the candidate method. For candidate methods which provide a direct measurement of lead concentrations without a separable procedure, $C_{1A} = C_{1B} = C_{1C}$, $C_{2A} = C_{2B} = C_{2C}$, etc.

(d) *Average lead concentration.* For the reference method, calculate the average lead concentration for each filter by averaging the concentrations calculated from the three analyses:

Equation 1

$$R_{i\,ave} = \frac{R_{iA} + R_{iB} + R_{iC}}{3}$$

where:

i is the filter number.

(e) *Acceptable filter pairs.* Disregard all filter pairs for which the lead concentration as determined in the previous paragraph (d) of this section by the average of the three reference method determinations, falls outside the range of 0.5 to 4.0 µg/m³. All remaining filter pairs must be subjected to both of the following tests for precision and comparability. At least five filter pairs must be within the 0.5 to 4.0 µg/m³ range for the tests to be valid.

(f) *Test for precision.* (1) Calculate the precision (P) of the analysis (in percent) for each filter and for each method, as the maximum minus the minimum divided by the average of the three concentration values, as follows:

Equation 2

$$P_{Ri} = \frac{R_{i\,max} - R_{i\,min}}{R_{i\,ave}} \times 100\%$$

or

Equation 3

$$P_{Ci} = \frac{C_{i\,max} - C_{i\,min}}{C_{i\,ave}} \times 100\%$$

where:

i indicates the filter number.

(2) If any reference method precision value (P_{Ri}) exceeds 15 percent, the precision of the reference method analytical procedure is out of control. Corrective action must be taken to determine the source(s) of imprecision and the reference method determinations must be repeated according to paragraph (c) of this section, or the entire test procedure (starting with paragraph (a) of this section) must be repeated.

(3) If any candidate method precision value (P_{Ci}) exceeds 15 percent, the candidate method fails the precision test.

(4) The candidate method passes this test if all precision values (i.e., all P_{Ri}'s and all P_{Ci}'s) are less than 15 percent.

(g) *Test for accuracy.* (1)(i) For the audit samples calculate the average lead concentration for each strip by averaging the concentrations calculated from the three analyses:

Equation 4

$$Q_{i\,ave} = \frac{Q_{iA} + Q_{iB} + Q_{iC}}{3}$$

where:

i is audit sample number.

(ii) Calculate the percent difference (D_q) between the indicated lead concentration for each audit sample and the true lead concentration (T_q) as follows:

Equation 5

$$D_{qi} = \frac{Q_{i\,ave} - T_{qi}}{T_{qi}} \times 100\%$$

(2) If any difference value (D_{qi}) exceeds ±5 percent, the accuracy of the

reference method analytical procedure is out of control. Corrective action must be taken to determine the source of the error(s) (e.g., calibration standard discrepancies, extraction problems, etc.) and the reference method and audit sample determinations must be repeated according to paragraph (c) of this section, or the entire test procedure (starting with paragraph (a) of this section) must be repeated.

(h) *Test for comparability*. (1) For each filter pair, calculate all nine possible percent differences (D) between the reference and candidate methods, using all nine possible combinations of the three determinations (A, B, and C) for each method, as:

Equation 6

$$D_{in} = \frac{C_{ij} - R_{ik}}{R_{ik}} \times 100\%$$

where:

i is the filter number, and n numbers from 1 to 9 for the nine possible difference combinations for the three determinations for each method (j = A, B, C, candidate; k = A, B, C, reference).

(2) If none of the percent differences (D) exceeds ±20 percent, the candidate method passes the test for comparability.

(3) If one or more of the percent differences (D) exceeds ±20 percent, the candidate method fails the test for comparability.

(i) The candidate method must pass both the precision test (paragraph (f) of this section) and the comparability test (paragraph (h) of this section) to qualify for designation as an equivalent method.

§ 53.34 Test procedure for methods for PM_{10} and $PM_{2.5}$.

(a) *Collocated measurements.* Set up three reference method samplers collocated with three candidate method samplers or analyzers at each of the number of test sites specified in table C–4 of this subpart. At each site, obtain as many sets of simultaneous PM_{10} or $PM_{2.5}$ measurements as necessary (see paragraph (c)(3) of this section), each set consisting of three reference method and three candidate method measurements, all obtained simultaneously. For $PM_{2.5}$ candidate Class II equivalent methods, at least two collocated PM_{10} reference method samplers are also required to obtain $PM_{2.5}/PM_{10}$ ratios for each sample set. Candidate PM_{10} method measurements shall be 24-hour integrated measurements; $PM_{2.5}$ measurements may be either 24- or 48-hour integrated measurements. All collocated measurements in a sample set must cover the same 24- or 48-hour time period. For samplers, retrieve the samples promptly after sample collection and analyze each sample according to the reference method or candidate method, as appropriate, and determine the PM_{10} or $PM_{2.5}$ concentration in $\mu g/m^3$. If the conditions of § 53.30(d)(4) apply, collect sample sets only with the three reference method samplers. Guidance for quality assurance procedures for $PM_{2.5}$ methods is found in section 2.12 of the Quality Assurance Handbook (reference 6 of appendix A to subpart A of this part).

(b) *Sequential samplers.* For sequential samplers, the sampler shall be configured for the maximum number of sequential samples and shall be set for automatic collection of all samples sequentially such that the test samples are collected equally, to the extent possible, among all available sequential channels or utilizing the full available sequential capability.

(c) *Test for comparability and precision.* (1) For each of the measurement sets, calculate the average PM_{10} or $PM_{2.5}$ concentration obtained with the reference method samplers:

Equation 7

$$\overline{R}_j = \frac{\sum_{i=1}^{3} R_{ij}}{3}$$

where:

R denotes results from the reference method;
i is the sampler number; and
j is the set.

(2)(i) For each of the measurement sets, calculate the precision of the reference method PM_{10} or $PM_{2.5}$ measurements as: